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From: Taryn Scholz

Date: March 1, 2007

To: Eric Pastor – Pastor, Behling & Wheeler, LLC

Re: Gulfco Superfund Site
R-flagged Analytical Results for Antimony in Soils

Dear Eric,

Several of the Antimony results for soils from the Gulfco site are R-flagged (unusable), indicating a need for further review of the data and an evaluation of the analytical technique. I have completed the review and evaluation and the findings are given below.

Summary of Sample and QC Results

The soil results for Antimony can be summarized as follows:

- The data set includes ten 'SS' (Lot 21 surface soil) samples plus one field duplicate from the Lot 21 area, of which all are non-detects. One MS/MSD pair was collected, prepared, and analyzed with the samples. The average MS/MSD recovery is low and <30%. All results (which are all non-detects) were R-flagged during the validation.
- The data set includes 27 'SB' (soil boring) samples plus two field duplicates from the North Area, of which twelve are non-detects. Two MS/MSD were collected (or selected by the laboratory for their internal QC), prepared, and analyzed with the samples. The average MS/MSD recoveries for Antimony are low but $\geq$30%. All results (both detects and non-detects) were qualified as estimated (with a low bias if above the quantitation limit, i.e. not B-flagged by the laboratory) during the validation.
- The data set includes 166 'SB' (soil boring) samples plus eight field duplicates from the South Area, of which 106 are non-detects. Thirteen MS/MSD were collected (or selected by the laboratory for their internal QC), prepared, and analyzed with the samples. The average MS/MSD recoveries for Antimony are low but $\geq$30% for four of the MS/MSD pairs and <30% for nine of the MS/MSD pairs with one pair at 0% recovery. One detect was qualified with a U-flag due to field blank contamination. The remaining detects were qualified as estimated (with a low bias if above the quantitation limit, i.e. not B-flagged by the laboratory). For the non-detects, 22 were qualified as estimated and 84 were R-flagged during the validation, depending upon the recovery of the associated MS/MSD. (Note that MS/MSD pairs were associated with samples that were collected during the same event (i.e., collected, shipped, and received at the same time) from the same area of the site (i.e., the North Area, the South Area, or the Lot 21 area) and, in all but a few cases, were prepared (i.e., digested) in the same batch.)

Table 1 shows the sample IDs and QC results for each MS/MSD pair.

Evaluation of Post Digestion Spike Results

When the MS/MSD recovery for a metal falls outside the control limits, some laboratories analyze a Post Digestion Spike (PDS) as required per CLP protocol (though not required per SW846 methodology). For the MS/MSD, the spike is added to the sample before preparation and is carried through the digestion and analysis. For the PDS, the spike is added to the sample after the digestion and is carried through the analysis.

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During the validation, Antimony results for non-detects associated with an MS/MSD recovery <30% were summarily rejected (i.e., R-flagged) without evaluation of any PDS results. This is consistent with Table 1 (Guidance for Assignment of Data Qualifier Flags) in SOP no. 16 for Data Validation in the Gulfco QAPP. However, per item (16) on page 7, the SOP also stipulates that professional judgment (and Post Digestion Spike results, if available) should be used to evaluate whether sample results associated with low-biased MS recoveries should be rejected or qualified as estimated concentrations with potiential low bias.

The laboratory did analyze a Post Digestion Spike for each deficient MS/MSD and reported the recoveries in the hardcopy laboratory packages. The PDS recoveries for Antimony in the soils are shown in Table 1. For every MS/MSD pair, the PDS shows good accuracy with a minimum recovery of 82% and a maximum recovery of 114%.

Data Usability

The purpose of the soil investigation is to evaluate the lateral and vertical extent of COIs in soils to evaluate potential human health and ecological risks associated with direct contact with and ingestion of soil, or potential runoff from these areas to surface waters (RI/FS Work Plan, Gulfco Marine Maintenance Superfund Site, 3/14/06, pg. 27).

According to the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, October 2004 (NFG), sample data associated with a Matrix Spike with low recovery should be qualified as follows:

> *If the Matrix Spike %R is <30%, verify that a post-digestion spike was analyzed if required. If the post-digestion spike %R is <75% or is not performed, qualify sample results that are ≥ Method Detection Limit (MDL) as estimated low (J-) and non-detects as unusable (R). If the post-digestion spike %R is ≥75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).*

According to the USEPA Guidance for Data Usability in Risk Assessment (pg. 113), data that are qualified as estimated according to the guidelines in the NFG should be used for risk assessment purposes.

Based on the above, the Antimony results for the soil samples, which were R-flagged during the validation based on the MS/MSD results, have been further evaluated taking into consideration the PDS results, and the data flagging has been updated using the following guidelines:

> If the MS or MSD %R is <30% but both are >0%, verify that the PDS %R is ≥ 75% and qualify detects as estimated with a low bias (J-) and non-detects as estimated (UJ). If the PDS %R is <75% or is not performed, qualify detects as estimated with a low bias (J-) and non-detects as unusable (R).
>
> If the MS and MSD %R is equal to 0%, qualify detects as estimated with a low bias (J-) and non-detects as unusable (R).

This is considered a conservative approach since (1) though not required per the NFG, data associated with a 0% MS/MSD recovery are rejected (to minimize likelihood of false negatives) and (2) though considered estimated with an unknown bias per the NFG, data associated with a low MS/MSD recovery but good PDS recovery are considered to be estimated with a low bias (since the recovery through the digestion is low). This is consistent with the approach used for organics analyses for the Gulfco site, for which data are also rejected if associated with a 0% MS/MSD recovery, though this is not required per the NFG.

The attached spreadsheet includes records for all affected Antimony results with updated Validation_Qualifiers and QC_Comments. The data set now includes six R-flagged results out of 214 total results giving a

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completeness of 97% for Antimony in soils. Note that some results for other metals in the first-round samples (i.e., for Antimony in sediments and for Tin in soils) have been summarily rejected (i.e., R-flagged) due to MS/MSD recovery <30% without evaluation of the PDS results. However, a relatively low number of results are R-flagged (7 of 64 Antimony results in sediments and 8 of 214 Tin results in soils), and thus these data have not been re-evaluated. PDS data will be evaluated for all future samples as noted below.

Corrective Action

The following corrective actions are being taken to improve data quality for future analyses:

1. The fact that the MS/MSD recovery is low while the PDS recovery is good suggests that the sample matrix may be difficult to digest. The laboratory is investigating alternate digestion techniques to improve recovery of Antimony in soils.

2. All validations will now include an evaluation of PDS recovery results for any metal with <30% recovery in the MS or MSD. Data will be qualified as detailed above.

If you have any questions, please do not hesitate to contact me.

Sincerely,

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TABLE 1
Gulfco Marine Maintenance Superfund Site
Summary of First-Round MS/MSD Results
for Antimony in Soils

Parent Sample_ID	Lab_Sample_ ID	Collection_ Date	Collection_ Time	Lab_ Receipt_ Date	Parameter	Prep_ Factor	Dilution_ Factor	Prep_ date	Prep_ time	Analysis_ Date	Analysis_ Time	MS Waived?	MS %R	MSD %R	Average MS/MSD %R	RPD	PDS %R
Residential Surface Soils																	
SA3SS03-003-(0-1)	20608045214	8/3/2006	8:40	8/4/2006	Antimony	40	1	8/7/2006	14:45	8/9/2006	14:56	no	12	13	12.5	14	98
Soil Borings from North Area																	
ND3SB05-010 (1-2)	20607191622	7/17/06	16:20	7/19/06	Antimony	40	1	7/21/06	12:05	7/22/06	17:23	no	36	44	40	16	99
NF3SB14-028-(1-2)	392868	7/20/06	11:50	7/21/06	Antimony	40	1	7/24/06	11:35	7/29/06	10:58	no	38	36	37	3	104
Soil Borings from South Area																	
SA1SB15-030-(1-2)	20607210603	7/19/06	16:45	7/21/06	Antimony	40	1	7/23/06	18:20	7/29/06	12:04	no	7	8	7.5	16	88
SC1SB25-050-(1-2)	20607210913	7/19/06	15:15	7/21/06	Antimony	40	1	7/25/06	13:40	7/31/06	13:23	no	4	3	3.5	55	88
SD3SB33-066-(1-2)	393051	7/19/06	16:35	7/21/06	Antimony	40	1	7/24/06	15:00	7/25/06	17:46	no	0	0	0	0	88
SD5SB35-070-(1-2)	20607201906	7/19/06	12:10	7/20/06	Antimony	40	2	7/24/06	16:15	7/26/06	12:31	no	35	48	41.5	30	91
SE5SB41-082-(1-2)	392859	7/19/06	11:00	7/20/06	Antimony	40	1	7/23/06	15:45	7/24/06	18:03	no	23	22	22.5	2	108
SF3SB45-090-(1-2)	20607200604	7/18/06	16:25	7/20/06	Antimony	40	1	7/23/06	17:00	7/26/06	12:58	no	22	19	20.5	14	110
SG3SB55-110 -(1-2)	20607201223	7/18/06	16:00	7/20/06	Antimony	40	1	7/25/06	11:45	7/31/06	15:37	no	7	9	8	24	87
SH6SB67-134-(1-2)	20607201206	7/19/06	9:20	7/20/06	Antimony	40	1	7/24/06	17:00	7/26/06	17:17	no	43	45	44	5	104
SJ3SB79-158-(1-2)	392162	7/17/06	16:45	7/19/06	Antimony	40	1	7/20/06	17:00	7/24/06	18:55	no	15	15	15	1	93
SJ5SB81-161-(0-0.5)	20607191433	7/18/06	12:20	7/19/06	Antimony	40	1	7/20/06	16:05	7/24/06	15:15	no	46	40	43	10	102
SJ6SB82-164-(1-2)	393912	7/18/06	12:50	7/19/06	Antimony	40	1	7/26/06	12:15	7/27/06	12:07	no	19	20	19.5	4	82
SK7SB91-181-(0-0.5)	20607191405	7/18/06	13:00	7/19/06	Antimony	40	1	7/20/06	12:05	7/24/06	13:14	no	0	12	6	200	91
SL8SB99-198-(1-2)	20607191626	7/18/06	9:25	7/19/06	Antimony	40	1	7/23/06	14:00	7/24/06	16:45	no	31	33	32	7	114